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HEAT-RESISTANT SAGGERS PRODUCED BY SLIP CASTING

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Heat-resistant saggers containing granular silicon carbide filler with particles up to 130 μm in size were obtained by slip casting in gypsum molds. The silicon carbide content varied from 30 to 55 wt.%. Prototype batches of saggers of different shapes were tested in gas-heated and mazut-heated kilns and exhibited good service properties, which made it possible to decrease the amount of reject articles in firing.

Saggers of various shapes and dimensions, which are widely used in porcelain firing, make it possible to produce high-quality articles and to eliminate contamination defects. The following requirements are imposed on saggers: they have to be highly refractory (over 1670°C), rather strong in the air-dry state and after firing, highly resistant to deformation under loading at high temperatures, highly heat-resistant, and rather homogeneous and dense. To avoid contamination of articles by sagger particles, the interior walls and bottom of the sagger are coated with a refractory grease [1].

The heat resistance of a refractory material is higher, the more homogeneous its structure, the higher its thermal conductivity, and the more uniform its thermal expansion. A very negative effect on the heat resistance of a material is produced by sharp changes in volume caused by polymorphic transitions of SiO_2 and some other oxides.

Worsening of the service conditions of saggers due to intensification of the firing process predetermines the choice of the basic materials for their manufacture in the near future: cordierite, mullite, silicon carbide, and, to a lesser extert, fireclay, although the latter material is still widely used at small factories.

In considering heat-resistance problems, a certain significance is attributed to the rate of crack propagation, which is a two-stage process. In the first stage, nucleation of rapidly propagating cracks in a dense homogeneous ceramic body is determined by the thermal-resistance coefficient. In the second stage, the rate of crack propagation is related to the nature of the ceramic microstructure. The evolution and arrangement of structural microcracks impede the propagation and growth of thermal cracks, i.e., "technological" microcracks obstruct the propagation of the "thermal" cracks [2].

It was established in practice that the introduction of a granular filler and an increase in its amount and grain size improve the heat resistance of the material. This is apparently related to the specific structure of such a refractory. In the course of firing, stresses arise at the boundary of the grog additive and the fine-grained sintering binder, which causes ruptures and, accordingly, microcracks. The presence of these microcracks and ruptures near filler grains imparts localized adhesion to the ceramic material at these sites, which creates an opportunity for local stress relaxation due to mutual displacement of individual structural elements. These ruptures and microcracks can be regarded as a barrier impeding the propagation of thermal cracks. Different methods of mixture treatment and molding can also significantly affect the heat resistance of the product.

Along with the physicochemical properties, the heat resistance is subject to the effect of the size and shape of the product and the heating and cooling conditions of the product in service or in testing. The size of the article determines the extent of the strain and, consequently, the magnitude of the stresses arising. The shape of the article affects the nonuniformity of arising local stresses that facilitate cracking of the product during temperature changes.

The fireclay content in high-chamotte mixtures produced by a semidry method with increased molding pressure attains 80%. Substitution of corundum for the fine fireclay fractions (10 – 20%) increases the sagger turnover by 30 – 50%. Its strength in this case increases 1.5 – 2-fold, and the softening point is raised from 1320 – 1360 to 1420 – 1430°C. A silicon carbide sagger is characterized by an increased and high (up to 48 – 49%) silicon carbide content. This sagger can function well under a load of 0.05 – 0.06 MPa in the absence of impacts and provided a certain gas medium is maintained: first, an oxidizing medium up to 1100°C and then a reducing medium up to 1400°C. Firing of porcelain articles satisfies these conditions [3]. A silicon carbide sagger is 2.5 – 3 times

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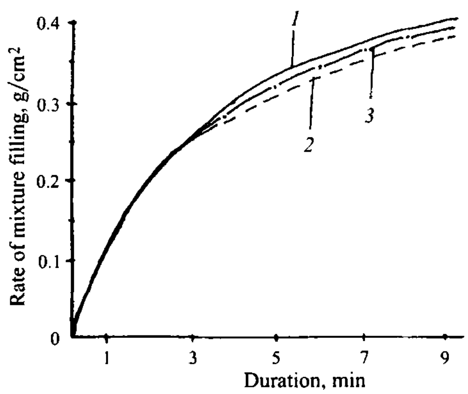

Fig. 1. Curves of filling a gypsum body with slip: *1*) industrial slip; *2* and *3*) KK-55M1 and KK-55M2 slips, respectively.

stronger than a fireclay sagger, which makes it possible to manufacture saggers with thinner walls and to increase the effective utilization of the kiln space. The high thermal conductivity of silicon carbide (8 times that of fireclay) makes it possible to shorten the duration of heating and cooling of fired articles and improve their quality.

Casting in gypsum molds is used to manufacture large-sized thin-walled saggers, mounts, and refractories of complex profile. Improvement of sagger material should be directed to increasing its service life and technological efficiency.

The choice of manufacturing technology is largely determined by the production and financial capacity of the company. Therefore, considering the material resources of the Gzhel' JSC, of all the various molding techniques the company was able to choose only between plastic molding of saggers on semiautomatic machines or casting in gypsum molds. Plastic molding of saggers made of fireclay mixtures with an addition of technical alumina had been used at the company until recently. This method allowed production of thick-walled round-shaped saggers with a low turnover (2 – 3 cycles on the average). Considering the continuing increase in prices for material, the production of these saggers no longer makes sense.

In this situation it was proposed that the turnover of saggers be increased by eliminating fireclay from the sagger mixture composition and introducing instead a material with a low TCLE, a high thermal conductivity, and a high resistance to the effect of kiln gases. Silicon carbide is precisely such a material, which makes it possible to produce a sagger with a turnover of up to 100 cycles or more, depending on the manufacturing technology.

A preliminary analysis of the sedimentation resistance of slips was carried out, since saggers of different sizes and shapes can be produced only by slip casting technology. An analysis of the sedimentation stability of slip that contained silicon carbide particles demonstrated the possibility of producing slip that is resistant to stratification for the prescribed period during which the gypsum mold is being filled with the mixture. At the same time, the slip should have high mobility and ensure high packing density of the particles in the cast piece. Preliminary experiments proved the possibility of using silicon carbide particles up to 130 μm in diameter for casting slip.

Slip mixtures that contained 55 wt.% silicon carbide with particle sizes of –125 and –200 μm were mixed in a ball mill with a previously prepared suspension that contained argillaceous materials and alumina. This method makes it possible to eliminate protracted milling of silicon carbide in a ball mill. However, it is not as universal as the joint-grinding method, which allows for use of almost any grade of silicon carbide powder (No. 40 and over), which is cheaper and more available.

The granular composition of the SiC powder (– 125 μm) is as follows: residue on a sieve with a 0.2 mm mesh size — none; 0.125 mm — 4.2%; 0.100 mm — 60.8%; 0.063 mm — 30.9%; below 0.063 mm — 4.1%. The granular composition of the SiC powder (– 200 μm) is as follows: residue on a sieve with a 0.25 mm mesh size — none; 0.200 mm — 1.5%; 0.125 mm — 71.0%, 0.063 mm — 23.5%; below 0.063 mm — 4.1%.

For comparison, fireclay-based slip traditionally used at the company was prepared by joint milling of its components up to a residue of 10 – 12% on a sieve with a 0.063 mm mesh size.

The rheological parameters of the slips are given in Table 1 (η_1 and η_2 are the viscosity after 30 and 210 sec holding).

The rate of mixture filling was determined by the gypsum crucible method; the resulting data are shown in Fig. 1.

Saggers were cast using casting in gypsum molds. The length of crock formation was assigned depending on the slip properties, moisture, and required thickness of the article wall. The wall thickness of the sagger should not exceed the wall thickness of the cast article by more than 2 or 3 times. Assuming this, the optimum duration of formation of crock 15 mm thick amounted to 65 – 75 min. After souring and straightening, the sagger was dried to a residual moisture of not more than 2%. The dried product was subjected to the first firing in a gas chamber furnace at a temperature of 970 – 980°C with a heating rate of 100 – 120°C/h and a hold for 1 h in an oxidizing medium.

TABLE 1

Slip grade	Rheological-test viscosity, Pa · sec		Structuring coefficient
	η_1	η_2	
KK-55M	0.33	1.08	3.27
KK-55M1	0.41	1.60	3.90
KK-55M2	0.27	0.67	2.47
KK-55/–125	0.35	1.15	3.30
KK-55/+125	0.43	1.46	3.40
KK-30	0.44	2.37	5.38
RSh-1	0.42	2.31	5.50

Next for the purpose of reducing the contamination and extending the service life of the sagger by decreasing the oxidation of the surface layers of silicon carbide, the sagger was engobbed with high-temperature grease based on clay and alumina. The second firing was carried out at a temperature of 1320°C with a hold for 2 h according to the porcelain firing conditions.

The moisture of the slips varied from 29 – 30% for a 55% silicon carbide content to 35 – 36% for a 30% content of silicon carbide and 35 – 36% for fireclay slip. Dried samples were tested for bending, and the bending strength varied from 1.0 to 2.5 MPa depending on the content and coarseness of the silicon carbide fraction. The larger the silicon carbide particles, the lower the sample strength. After the first firing, the bending strength of the samples was 3.7 – 9.0 MPa. After the second firing, the bending strength attained 17 – 44 MPa.

Using KK-55M1 and KK-55M2 slips, trial batches of saggers with walls 5 to 9 mm thick were prepared: 4 round saggers 270 mm in diameter and 80 mm high, and 37 elliptical saggers 55 mm high with a major axis of 410 mm.

Two round saggers were cast from KK-55/+125 and KK-55/–125 slips each, and one round sagger and a tray were made from KK-55M slip.

15 elliptical and 2 round saggers were sent for testing in a mazut-heated kiln; 20 elliptical and 4 round saggers were tested in a gas kiln, and 5 round and 2 complex-shaped saggers and a tray were tested in a chamber furnace.

The firing conditions in the gas kiln and the chamber furnace differ in their heating and cooling conditions, which are "milder" in the chamber furnace. In turn, the firing conditions in the mazut kiln are "milder" than in the chamber furnace. Rapid cooling from 1000°C in the gas kiln produces high thermal stresses inside the sagger and brings about its premature removal from circulation. Moreover, the silicon carbide sagger requires careful handling due to its brittleness.

Under these conditions the fireclay sagger has an average turnover of around 2 – 3 cycles.

TABLE 2

Firing	Rejected items, %		
	after 4 firing cycles	after 11 firing cycles	after 16 firing cycles
In the mazut kiln	None	6	None
In the gas kiln	10	21	37

Test results of cast products containing silicon carbide are given in Table 2.

The tray and the round sagger made of KK-55M slip performed 54 cycles in the chamber furnace with a very "mild" procedure. The saggers made of KK-55/+125 and KK-55/–125 slips made 47 cycles and are still in service.

Thus, the development of a heat-resistant sagger containing silicon carbide is most promising with respect to saving material resources, prolongation of service life, and improvement of the fired-article quality. The resulting products that contain silicon carbide exhibit good service characteristics: strength, heat resistance, low shrinkage, etc.

Implementation of the technology of sagger production by slip casting makes it possible to manufacture long-lasting saggers of virtually any dimensions, which is important in the production of small-run and unique articles.

The tested batch of saggers made of KK-55M1 and KK-55M2 slips showed good service properties.

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